The Spokesman

FEBRUARY • 1952

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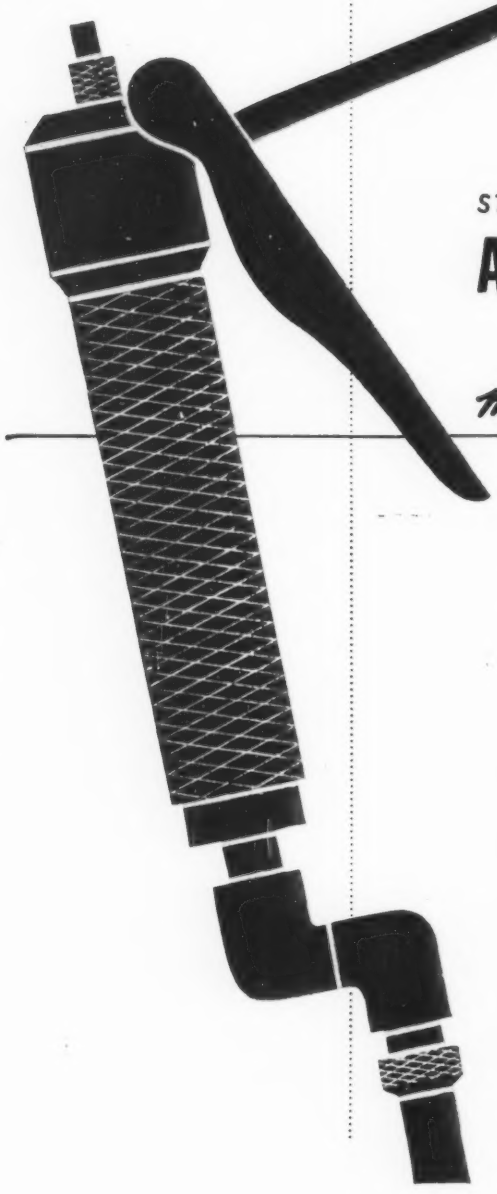

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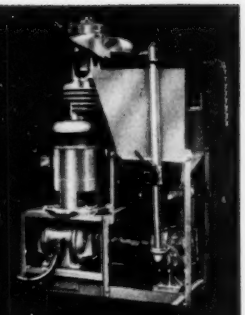

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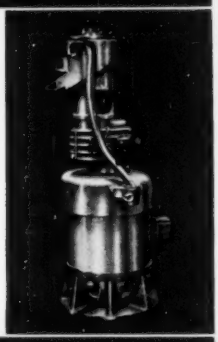

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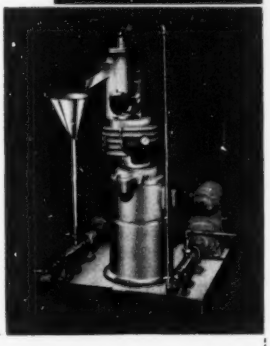

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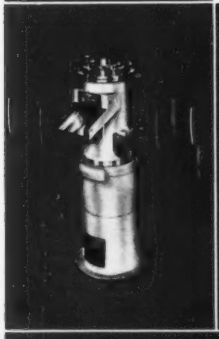

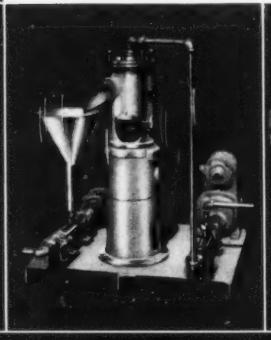

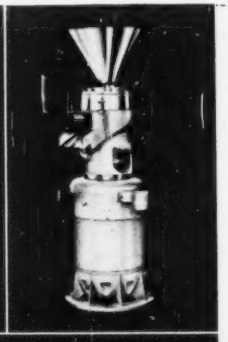

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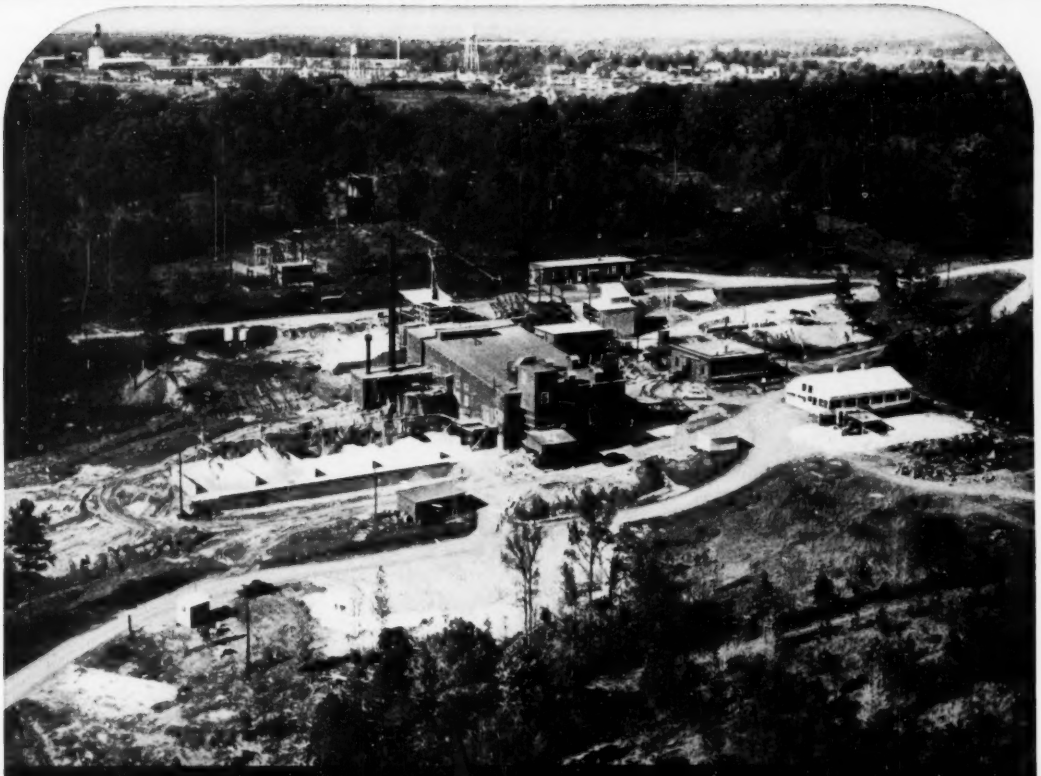

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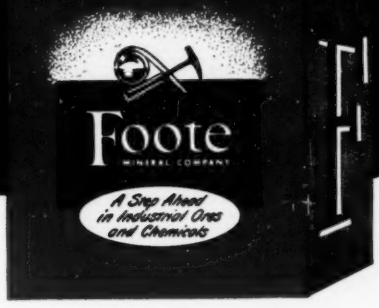

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ABOUT THE COVER

Featured on the cover is one of several stainless steel reaction kettles used in the Witco semi-works unit. In this pilot-plant operation, reactions are carefully studied to insure selection of optimum conditions for plant production of new stearates. Fifty-pound samples or larger lots can be prepared for submission to prospective users.

The photo-inset shows the enlarged Chicago laboratories of the Witco Chemical Company. The new space comprises a two-story wing, added to the former laboratory building, which provides an additional working area of 6,500 square feet. The space, already occupied, is devoted to research in the development of new and improved materials for use in the field of lubricating greases as well as in other industries.

Witco has effectively utilized its research facilities and has developed aluminum and lithium soaps which are reported to facilitate the production of uniform, economical greases of high quality. Lead soaps of the type exhibiting excellent extreme pressure and anti-corrosion properties have been developed for use in gear oils, and work has recently been undertaken leading toward the production of synthetic di-esters.

The Witco laboratories are located on a 17-acre tract near the Chicago airport, along with one of Witco's four chemical plants.

President's page

by George E. Merkle, President, N.L.G.I.

LISTENING TO REASON

As I ponder over subjects suitable for this page, many present themselves as timely and important, not to one nor a few groups but to the peoples of the world.

It is difficult to treat such subjects without appearing to attempt to preach. Preaching is good but I will leave this to those better suited to the task than I.

We have all listened to the prolonged bickering in the United Nations meetings and in the so-called Peace Meetings between the U. N. and Communists over the Korean situation. We also hear and read of endless such discussions over many domestic issues. Some of the differences may be considered justified and subject to debate, but we are all aware of the annoying differences due to unwillingness to consider the other fellow's views and rights. This latter condition could be avoided if everyone were willing to "Listen to Reason."

It is difficult for peoples of nations whose education and governments are very different to understand each other, but with reasoning entering the picture most certainly much more progress could be made and we would all be much the happier and better for it.

One of the outstanding examples of reason, cooperation and intelligent understanding is the N L G I. No one has a selfish motive. All work for the good of the group without fanfare. The good fellowship in this organization is better than often found in fraternal organizations. It could hardly be better and certainly sets a good example for opposing or competing factions, for in our Institute the members are actually in competition with each other in most instances. This fine example should be copied by others.

LITHIUM

Detailed Study By

JOHN A. BROWN, CHARLES N. HUDSON
LEWIS D. LORING

USE OF THE ELECTRON MICROSCOPE IN THE EXAMINATION OF GREASES

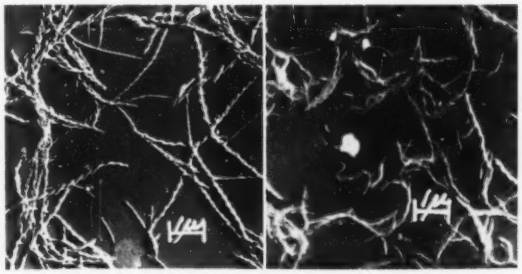
In recent years the electron microscope has become a very useful and important tool in the study of lubricating greases, and a number of workers have used this tool to explore various grease problems. (1, 3, 4, 9, 11, 12, 14) Detail of the soap fiber structure can be obtained by no other means in use at the present time. The resolution of the optical microscope is not sufficient to reveal the detail of the soap fiber structure. X-ray diffraction gives information about unit crystals in detail much finer than the size of the fibers. The electron microscope fortunately is able to bridge the gap and to reveal the fine detail of the fiber structure.

The technique of preparing specimens for the electron microscope (7, 12) is somewhat different from that used in optical microscopy since specimens and specimen supports must be very much thinner for electron microscopy.

The procedure used in our laboratory for preparing specimens is as follows: A section of specimen screen (200 mesh to the inch) is covered with a thin film of formvar (an organic plastic material) as is normally done in electron microscopy. A small amount of grease is vigorously agitated in a large amount of ASTM naphtha to give a fairly dilute dispersion. One or two drops of this suspension are placed on the specimen screen carrying the plastic film and allowed to dry. The preparation is then washed with ASTM naphtha. The specimen in this condition would not provide any detail if viewed directly on the electron microscope since there is not enough contrast inherent in the soap itself. Therefore, a technique called shadowcasting is used, (15) in which a very thin film of a heavy metal is evaporated on to the specimen at an oblique angle, and the sample is then examined in the electron microscope. This method enhances detail on the fiber surfaces and also provides a permanent shadow from which information about the third dimension of the sample can be obtained. Chromium metal is used in our laboratory for shadowing purposes since it gives consistently good results with a smooth background and does not become granular in the manner of gold and some other metals when subjected to the electron beam in the microscope.

Some doubt has been raised as to whether the fiber structure revealed by this method is the true structure or whether it has been modified in the sample preparation process. Several experiments indicate that the process is a legitimate one and that the fiber structure revealed by the electron microscope is the true structure.

In order to prove this point a lithium grease was examined by spreading a very thin film of grease on to a specimen support and shadowing without use of naphtha at any time. The fiber structure, although poorly resolved, (Fig. 2) was shown to be the same as that obtained by the solvent leaching method (Fig. 1). (Magnification marks on all micrographs in this paper are equal to 1 micron (μ) which is



NAPHTHA LEACH
FIG. 1

NO NAPHTHA LEACH
FIG. 2

ABOVE is shown the entrance to the Sinclair Research Laboratories, Inc., at Harvey Illinois.

GREASE

Electron Microscope

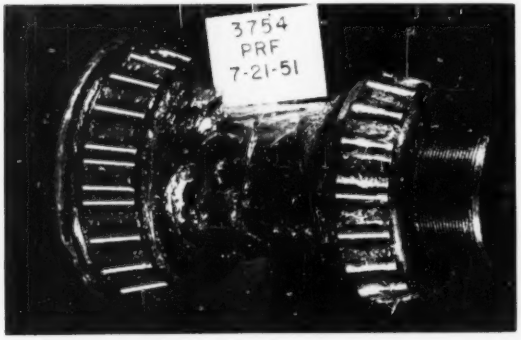
SINCLAIR RESEARCH LABORATORIES, INC.
HARVEY, ILLINOIS

.0001 cm. in length. Also 1 Angstrom unit (A) is .00000001 cm.).

X-ray diffraction examination shows that the crystal structure of the soap is the same whether in the oil environment as in a grease or in the leached state free of oil as when prepared for examination in the electron microscope. While this is not positive proof that the fibers themselves maintain the same size and shape in the leached condition that they had in the grease, it suggests that this is the case.

Greases which have been made by different methods but from the same components sometimes show completely different structures when examined by the electron microscope. Chemically these greases are the same, the only difference being that of the physical state of the soap component. Hence, the electron microscope reveals differences where chemistry does not. This is an important application of the electron microscope to grease problems since it enables one to examine the effects which process variables, testing procedures, and actual mechanical work have on the fiber structure.

These data indicate that the electron microscope is a useful and legitimate tool for grease examination and that the method of preparation has not appreciably altered the fiber structure.



GREASE STRUCTURE—OPTICAL BIREFRINGENCE—ORIENTATION

The optical microscope as well as the electron microscope has aided in revealing the bulk structure of greases. It is generally known that greases are colloidal systems of solid soap fibers and liquid oil. These systems set up into a gel structure with certain definite rheological properties. It is this gel structure which is worthwhile investigating since it largely determines the physical characteristics of the bulk grease.

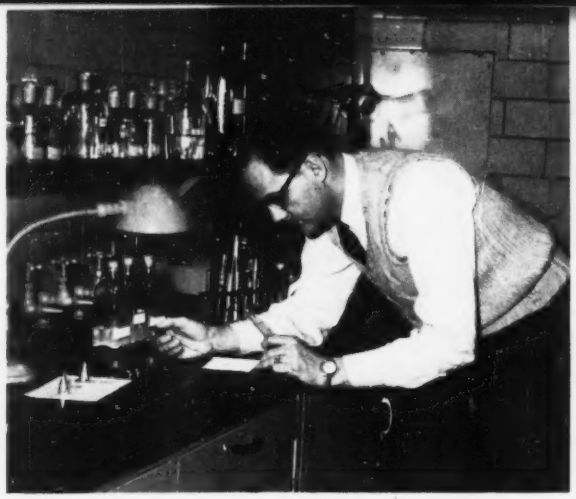
If a grease is spread onto an ordinary microscope slide and is then examined under polarized light, it is noted that the whole grease mass acts as a single crystal of a double refracting or birefringent nature. This phenomenon of birefringence is exhibited by any crystal which has two different indices of refraction along the different crystallographic axes. All but the simple cubic crystals possess this property. Birefringence is detected by examining the material under polarized light between two crossed Nicol prisms. As the crystal is rotated in polarized light it becomes alternately light and dark. This is a common microscopic method of crystal examination. (6) Crystals of this sort in a random state would then show some crystals light while others were dark at the same time. If the crystals were all aligned in a field, then all the crystals would be light simultaneously or dark simultaneously as they are rotated in polarized light.

Since soap fibers are crystals in the true sense of the word with different crystal axes, they will also react the same in polarized light. If now these fibers are randomly situated some will be dark while others are light. If they

---

ABOVE . . . . John A. Brown sits at his desk in a corner of the electron microscope laboratory.
LEFT . . . . Lithium grease lubricates truck wheel bearings.

1 FORMVAR FILM will cover the specimen screen used in the electron microscope. John A. Brown prepares the film which will give tranparent support.



2 A SAMPLE IS MOUNTED on the formvar-covered specimen screen for the electron microscope. The grease sample is dispersed in naphtha and one or two drops of this suspension is placed on the formvar-covered screen.

A Grease Specimen Is Prepared For the Electron Microscope

ABOVE . . . . OPPOSITE PAGE: Co-author John A. Brown shows the steps used in the process of grease specimen preparation.

are all oriented in the same direction they will all be light simultaneously or dark simultaneously. Since the whole mass of grease spread out on a slide becomes completely light or dark at once it indicates that all the soap fibers are oriented in the same direction. If the grease now is spread at some other angle to the first spread, the orientation of all the fibers then takes the direction of the new spread. The ease with which this orientation is accomplished is somewhat surprising. A single spreading stroke is sufficient to cause complete orientation.

This orientation helps to elucidate grease structure. It helps to show that the soap fibers form a structure by themselves when in grease form and that they will maintain this form when the oil is leached out. (5)

The following experiment indicates this to be true.

A sample of grease was spread onto a glass microscope slide. This sample showed strong birefringence. The grease film was immersed in a beaker of quiescent ASTM naphtha to leach out the oil without disturbing the soap film. After removal from the naphtha the dry soap film had the same birefringence as the original grease. A second sample of the grease was vigorously shaken with naphtha to disperse the fibers completely, and a few drops of this suspension were dried on a microscopic slide. This random suspension showed no oriented birefringence. This proved that the fibers in the undisturbed soap film were the same as they were in the grease and that they were oriented in both cases. The random dispersion destroyed this orientation.

Birefringence in this grease system is similar to a phenomenon in colloid systems known as "streaming double refraction" whereby particles, such as rods or fibers suspended in a liquid, will align themselves in a flowing system so that their long axis is parallel to the direction of flow.

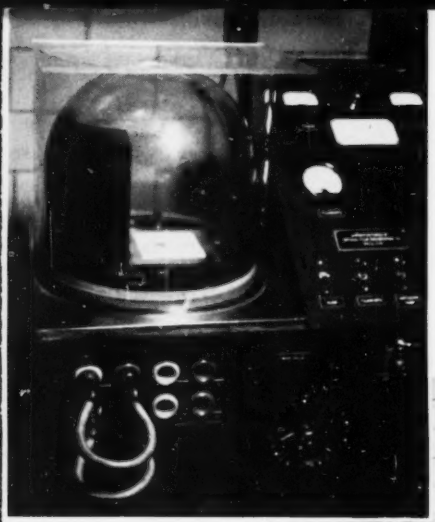
In the grease system the same orientation effect takes place (8, 10) except that it remains as a permanent effect after the orienting force is removed, whereas the colloidal system reverts to a random state when flow ceases.

A number of different greases with different fiber structures have been examined for this optical birefringence. In almost all cases greases that have well defined fibers as shown on the electron microscope gave strong oriented birefringence while greases with no apparent fibers gave little or none.

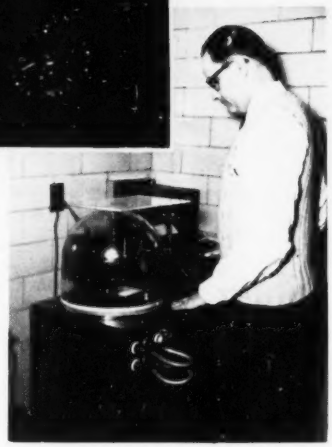
Some correlation has been noted between birefringence and colloid milling. In some cases a grease made without use of the colloid mill has not exhibited birefringence, but after treatment in the colloid mill the same grease now shows strong birefringence. This is strong proof that much fiber dispersion and orientation occurs in the colloid mill, especially since the individual fibers did not show any great change between the milled and unmilled sample as examined on the electron microscope.

The orientation of fibers previously mentioned can also be observed directly by examination on the electron microscope. A very thin film of grease spread onto a specimen mount was leached in ASTM naphtha without disturbance and shadowed. This preparation when examined on the electron microscope showed orientation of fibers in one predominant direction over the whole area of the specimen. Representative pictures of two oriented greases are as follows: (Fig. 3 and 4)

The arrangement of soap fibers in a grease and the reaction



Grease specimen is shadowed in an evacuated bell jar.



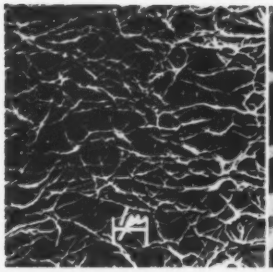
3 SHADOW CASTING a specimen of grease is demonstrated by Mr. Brown. A particle of chromium is heated by a tungsten wire so that it evaporates and forms a very thin directional coating on the sample.

4 CUTTING A DISK of specimen screen for use in the electron microscope, Mr. Brown uses a small punch similar to a cork borer. A large piece of screen was used in the preparation of the specimen.

5 THE DISK of specimen screen with the specimen on it is placed in the sample holder for use in the electron microscope.

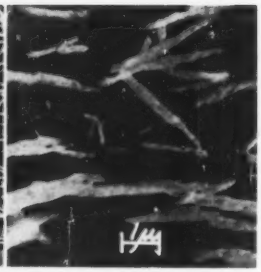
6 LEFT . . . . THE SAMPLE HOLDER is placed in the electron microscope.

7 RIGHT . . . . THE SPECIMEN IS STUDIED by the electron microscope. The image is viewed on a fluorescent screen similar to the screen in a television picture tube.



ORIENTED LITHIUM GREASE

FIG. 3

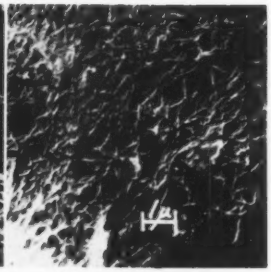


ORIENTED CALCIUM-SODIUM GREASE

FIG. 4

LITHIUM GREASE FROM 12 HYDROXY STEARIC ACID (320° F.)

FIG. 5



LITHIUM GREASE FROM THE GLYCERIDE OF 12 HYDROXY STEARIC ACID (320° F.)

FIG. 6

of these fibers in a moving system is thus revealed by use of both the electron microscope and optical microscope.

COMPOSITION VARIABLES IN LITHIUM GREASES

The effect of composition of a grease on its fiber structure is one of major consideration. An important composition variable is the fat or fatty acid component. In order to study the effect of using various fatty acids a series of lithium greases was made using 12 hydroxy stearic acid, the methyl ester of 12 hydroxy stearic acid, the glyceride of 12 hydroxy stearic acid (hydrogenated castor oil) and the polymerized form of 12 hydroxy stearic acid. Greases were prepared from these four materials in the same way at 320°F. maximum processing temperature with the following results. The grease from the acid is seen to exist in granular form with a very few primitive fibers. (Fig. 5)

The grease from the glyceride of 12 hydroxy stearic acid had short (μ) loosely twisted fibers as shown in the following picture. (Fig. 6)

The grease from the methyl ester of 12 hydroxy stearic acid also contained small loosely twisted fibers which were similar to the type found in the glyceride grease. (Fig. 7)

Finally the grease from the polymerized 12 hydroxy stearic acid contained very fine filament like fibers about (1 μ) long with a slight twist. (Fig. 8)

The penetrations of these four greases with a soap content constant at 10% are shown in the table below.

These data indicate that the larger fibers give the most rigid gel structure while the grease with granular soap particles has a very weak gelling effect at the maximum processing temperature of 320°F.

Lithium greases from oleic acid and stearic acid were also examined. The grease from oleic acid at 320°F. showed very long (10 μ or more) string-like fibers with no twist. (Fig. 9)

The grease made from 97% pure stearic acid at 320°F. shows small flat granular particles (ca. 1 μ). (Fig. 10)

A grease made from tallow at 320°F. shows fibers of intermediate length (ca. 3 μ) without any twisting characteristics. (Fig. 11)

These pictures show the wide variation of soap fiber structures obtained by the use of various fats and fatty

GREASE PENETRATION TABLE

Fat or Fatty Acid	U.W.Pen.	Worked Pen.(60)	Fiber Structure
Glyceride of 12 OH St. Acid	280	284	1 μ long 700A thick-loosely twisted
Methyl Ester 12 OH St. Acid	295	298	1 μ long 700A thick-loosely twisted
Polymerized 12 OH St. Acid	339	361	1 μ long 300A thick-moderately twisted
Free 12 OH St. Acid	403	446+	Granular particles (1/4 μ)

LITHIUM GREASE FROM METHYL ESTER OF 12 HYDROXY STEARIC ACID (320° F.)

FIG. 7

LITHIUM GREASE FROM A POLYMERIZED 12 HYDROXY STEARIC ACID (320° F.)

FIG. 8

LITHIUM GREASE FROM OLEIC ACID (320° F.)

FIG. 9

LITHIUM GREASE FROM 97% PURE STEARIC ACID (320° F.)

FIG. 10

acids in the preparation of the grease. These greases will understandably have widely different properties which may be chosen for a particular application.

EFFECT OF TEMPERATURE ON FIBER STRUCTURE

As in the case of all crystal systems the effect of temperature on particle formation in greases is very important. Fiber formation is aided by heating to a temperature near the solution point where well defined fibers are able to be formed, much as single crystals are grown in inorganic systems.

A grease was made from 12 hydroxy stearic acid at 320°F. The soap then existed as granular particles about ¼ micron in size. This grease was heated to 370°, 380°, 390°, and 400°F. A few primitive fibers were formed at the lower temperatures but at 400°F. perfectly twisted fibers of 10 μ or more in length were formed. Micrographs are as follows: (Fig. 12, 13, 14 and 15).

At 400°F. a pronounced gelling of the grease occurred in correlation with the formation of the long fibers. The grease changed from a No. 1 grade to about a No. 3 grade at this transition point.

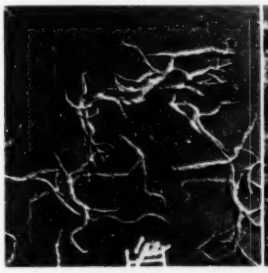
X-ray evidence of this phase transition indicates that it occurs at a somewhat lower temperature. Perhaps the X-ray method stresses the primitive fibers formed at the lower temperature more than the granular particles.

If the grease with the symmetrical form of twisted fiber is heated to its true solution point and cooled in air to room temperature the homogeneity of the fibers as well as the twist is destroyed. (Fig. 16)

These data indicate the important changes in the soap fiber structure due to temperature variations and the accompanying change of gelling power which the formation of fibers imparts to the grease.

EFFECT OF OXIDATION ON FIBER STRUCTURE

Oxidation of a grease in use has an adverse effect on its characteristics. It was desired to know if the soap fiber structure was affected in this oxidation, since this would

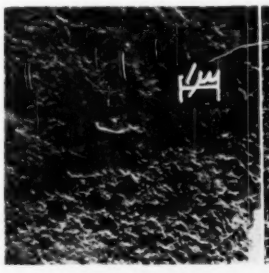


LITHIUM GREASE FROM BEEF TALLOW (320° F.)

FIG. 11

GREASE HEATED TO 370° F.

FIG. 12



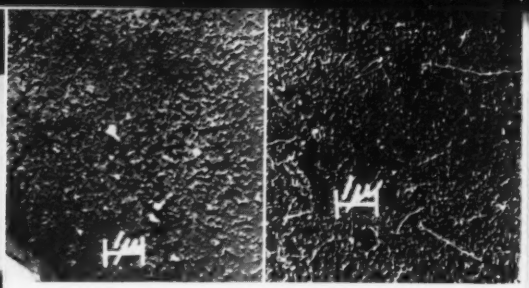
GREASE HEATED TO 380° F.

FIG. 13

GREASE HEATED TO 390° F.

FIG. 14

LEFT . . . . THE PENETROMETER is being used on a sample of grease to determine its consistency by Howard Gower. Virgin and used samples are run on this instrument to determine any change which occurs after working a grease.



VIRGIN LITHIUM GREASE AFTER 377 HOURS IN WHEEL BEARING

FIG. 19

LITHIUM GREASE AFTER 30 MINUTES IN WHEEL BEARING

FIG. 20

GREASE HEATED TO 400° F.

FIG. 15

LITHIUM GREASE FROM 12 HYDROXY STEARIC ACID TO 425° F.

FIG. 16

LITHIUM GREASE AFTER 177 HOURS NORMA HOFFMAN OXIDATION TEST.

FIG. 17

VIRGIN LITHIUM GREASE

FIG. 18

be of major concern. The following experiment was performed for this purpose.

A lithium grease with a well defined fiber structure as shown in Fig. 1 was oxidized in a Norma Hoffman bomb for 177 hours in presence of a copper catalyst. This grease showed no soap fibers remaining after oxidation. (Fig. 17) Some amorphous material was evident on the microscope but the fiber structure had vanished completely. The physical appearance was that of a liquid containing some black amorphous material. This shows that the soap fiber structure is adversely affected by oxidation, and illustrates the need for good inhibition of such greases. The above experiment is an example of accelerated oxidation. A study of the effect of progressive oxidation upon soap fiber structure is currently under way.

BREAKDOWN OF FIBER STRUCTURE WITH MECHANICAL WORK

One of the important aspects of grease research is a knowledge of the effect which severe mechanical working has on the grease fiber structure. If this structure is drastically changed as a grease actually lubricates the bearing surfaces, it is logical to assume that the bulk property of that portion will also be changed.

It was desired to examine a specimen of grease taken directly from the surface of a roller in a roller bearing, since this would give a good sample of grease which had actually been sheared between bearing surfaces. The method finally devised to obtain such a specimen was to take a piece of specimen screen covered with a formvar film and roll it over the surface of the roller without allowing it to contact the adjacent bulk grease. Some of the grease on the roller surface would be transferred to the formvar film. This sample could then be leached with ASTM naphtha, shadowed and viewed on the microscope.

This method was used on several greases in wheel bearing tests. In the first sample thus examined it was found after 377 hours performance in a laboratory wheel bearing that

VIRGIN LITHIUM GREASE

FIG. 21

1 PASS THROUGH COLLOID MILL

FIG. 22

RIGHT . . . FROM THE ROLLERS of a wheel bearing tester Charles N. Hudson takes a sample of grease. The tester provides a sample of grease which is broken down severely.

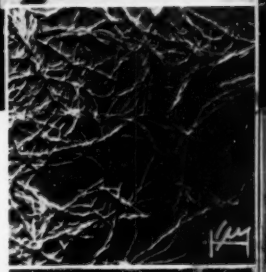
fibers which had been several microns long and twisted in the virgin grease (Fig. 18) had now degenerated into granular particles less than 700 A in size (Fig. 19) This was a reduction of size by a factor of about 50 or so. Very few fibers remained in their original form.

It was then desired to know at what time such destruction of fibers occurred. Further experiments indicated that complete degradation of fibers on the bearing surface could take place in a wheel bearing in less than 30 minutes. (Fig. 20)

The question then arose as to whether this degeneration was caused by excessive local heat or mechanical fracture. This was answered in the following way. A grease which has symmetrical twisted fibers in the virgin state shows granular particles with a few fibers after use in the bearing. The remaining few fibers still possess their characteristic twist although they are now much more thin than in the unworked state. Samples of this virgin grease which have been overheated to the solution point have lost the symmetrical twisted fibers and the fibers are now heterogeneous in size and shape. Therefore, the residual fibers in the worked specimen probably have not been overheated since they retain their characteristic shape.

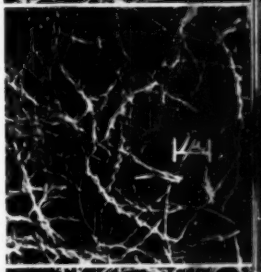
Also in this respect is the consideration of the effect of temperature on the soap in granular form previously described. If a granular low temperature grease is heated above its transition temperature it becomes fibrous in nature. But in the grease worked in a roller bearing the soap exists in granular form, after having been in fibrous form in the virgin state. It is logical to assume that if these granular particles had been subjected to a higher temperature they would have been reformed into fibers of some sort. Since this is not the case it is some indication that fiber destruction in this instance is not caused by excessive local temperature and would be due to mechanical fracture.

In the micrographs of some used greases, abrupt fracture edges can be seen at the fiber ends. This also is some proof that reduction of fiber length by work is a result of mechanical fracture.



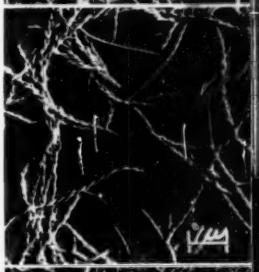
1 HOUR RECIRCULATION IN COLLOID MILL

FIG. 23



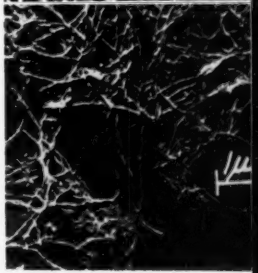
ABOVE GREASE AFTER 100,000 STROKES ASTM WORKER

FIG. 24



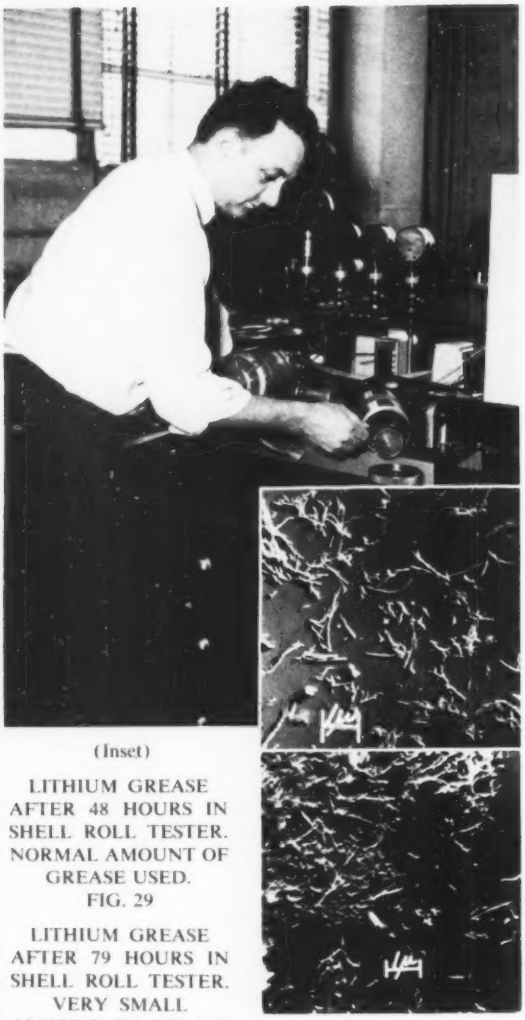
VIRGIN GREASE

FIG. 25



AFTER 100,000 STROKES IN NAVY WORKER

FIG. 26



(Inset)

LITHIUM GREASE AFTER 48 HOURS IN SHELL ROLL TESTER. NORMAL AMOUNT OF GREASE USED. FIG. 29

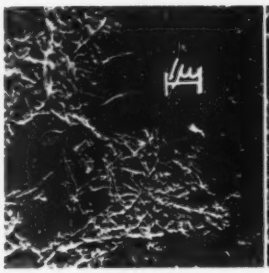
LITHIUM GREASE AFTER 79 HOURS IN SHELL ROLL TESTER. VERY SMALL AMOUNT OF GREASE USED. FIG. 30

RELATION OF VARIOUS WORK TESTS AND ACTUAL BEARING USE

Some attempt has been made to evaluate the intensity of work which the various work tests and bearing use perform on the grease soap fibers.

A lithium grease with homogeneous symmetrical twisted fibers was used in these tests since modifications in the fibers could be easily detected, (Fig. 21). This grease was worked in the ASTM worker, the Navy worker, a colloid mill, the Shell roll tester and a wheel bearing tester with the following results.

After one pass through the colloid mill at 0.003" clearance there was no appreciable effect on the fibers, (Fig. 22).



AFTER 300,000 STROKES IN NAVY WORKER FIG. 27

AFTER 500,000 STROKES IN NAVY WORKER FIG. 28

LEFT . . . . LEWIS D. LORING samples grease from the Shell Roll Tester, one of the workers described in this article.

After one hour of continuous recirculation through the mill the fibers had lost much of their twist and some fiber breakdown was noted, (Fig. 23).

After 100,000 strokes in the ASTM worker with ¼ inch holes the same grease as above showed that some of the fibers had lost their twist and some breakdown of fiber length was noted, (Fig. 24).

The broken fibers in this grease have very abrupt fracture edges indicating that this is a mechanical fracture of fibers.

Several tests were made on the Navy worker with 1/16 inch holes at increments of 100,000 strokes. After the first 100,000 strokes the soap fibers show somewhat more change than after 100,000 strokes in the ASTM worker, (Fig. 26). The fibers have been fractured more severely than in the ASTM worker after 100,000 strokes, with some particles being formed (ca. 2000 A). Many of the fibers have become thinner than normal and the whole soap structure is now more heterogeneous than normal in size and shape.

ASTM WORKERS are used on samples of grease by M. M. McCormick.

The same grease was examined after each additional 100,000 strokes. A progressive deterioration was noticed in that the number of small particles increased, the fibers lost much of their twist and the fiber structure in general became more heterogeneous, (Fig. 27 and 28).

Experiments were also run on the Shell roll tester using the same virgin grease as used in the ASTM and Navy worker tests. In one test the normal amount (70 grams) of grease was used. After 48 hours in the tester the fibers had been broken down to less than half their normal length. Some small particles had been formed but most of the fibers were homogeneous as to length, (Fig. 29).

In another test on the Shell roll tester a very small amount of grease was used. This amount was just sufficient to cover the roller with a thin film of grease. After 79 hours in the tester the fibers had been broken down severely, (Fig. 30). Many small particles were formed and the remaining fibers were less than half their normal size. The soap particles in this test were more heterogeneous in size and had lost more of their twist than those in the first test, where more grease was used.

These results indicate the relative severity of the various work tests. None of these tests, however, can match the destructive effect on the fibers which is found in actual roller bearings.

The important information derived from these experiments is that the mechanical work performed by the ASTM worker and Navy worker, etc., does not affect the fiber structure nearly as much as actual service conditions. Even 500,000 strokes in the Navy worker with 1/16 inch holes does not have the destructive effect of the wheel bearing.

SUMMARY

Use of the electron microscope as a tool in grease research has revealed the following interesting information about lithium greases:

1. The electron microscope indicates, in conjunction with optical microscope examination, how greases with well-defined fibers will orient their fibers in the bulk grease in a moving system.
2. The formation of long fibers in certain lithium greases when heated to the transition temperature is accompanied by pronounced gelling effect.
3. There are wide variations in the fiber structure of lithium greases depending on their composition. There appears to be some correlation between fiber structure as revealed by the electron microscope and physical and performance properties.
4. Certain lithium greases with well-defined fiber structures have very good mechanical stability as determined by long time tests in several types of laboratory working devices. Some of these greases when run in a wheel bearing can have their fiber structure completely destroyed in a very short period of residence on the actual bearing surfaces, which is accompanied by softening of this portion of the grease. If the fiber structure of the grease is such that the bulk of the grease is drawn through the bearing surfaces, softening of all the grease in the bearing assembly can occur to a much greater extent than would be predicted by laboratory worker tests.

The information gained thus far in this preliminary investigation of greases by the electron microscope has given an insight into the true structure of greases. This knowledge is of great value in the formulation of new and better greases, and the electron microscope has thus established itself in our laboratories as an important tool in grease research.

ACKNOWLEDGMENT

The authors wish to extend their thankful appreciation to Messrs. M. M. McCormick and H. D. Gower for the preparation of the experimental greases; to R. A. Van Nordstrand for the X-ray diffraction work; and to R. H. Gardner, M. Fairlie and J. W. Teter for their very welcome encouragement and direction during the course of this work.

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For appearance's sake, the sign is almost totally enclosed. As a result, considerable heat develops and failure of the motor's sintered-bronze bearings is common. Once, in fact, an entire order of 1000 signs was replaced less than a week after shipment because of bearing failures.

Then, 16 months ago, Flash-O-Graph ran comparative tests between six signs lubricated with the highest grade petroleum oils and six with Dow Corning 550 silicone oil. The best of the petroleum lubricated group lasted only 20 days without relubrication. All six of the silicone lubricated group are still running . . . without relubrication.

About a year ago Flash-O-Graph standardized on Dow Corning 550 silicone oil for the bearings of sign motors. They estimate their service problem has been reduced 99%. Of thousands of silicone lubricated units since put into the field, not one has been returned because of bearing failure.

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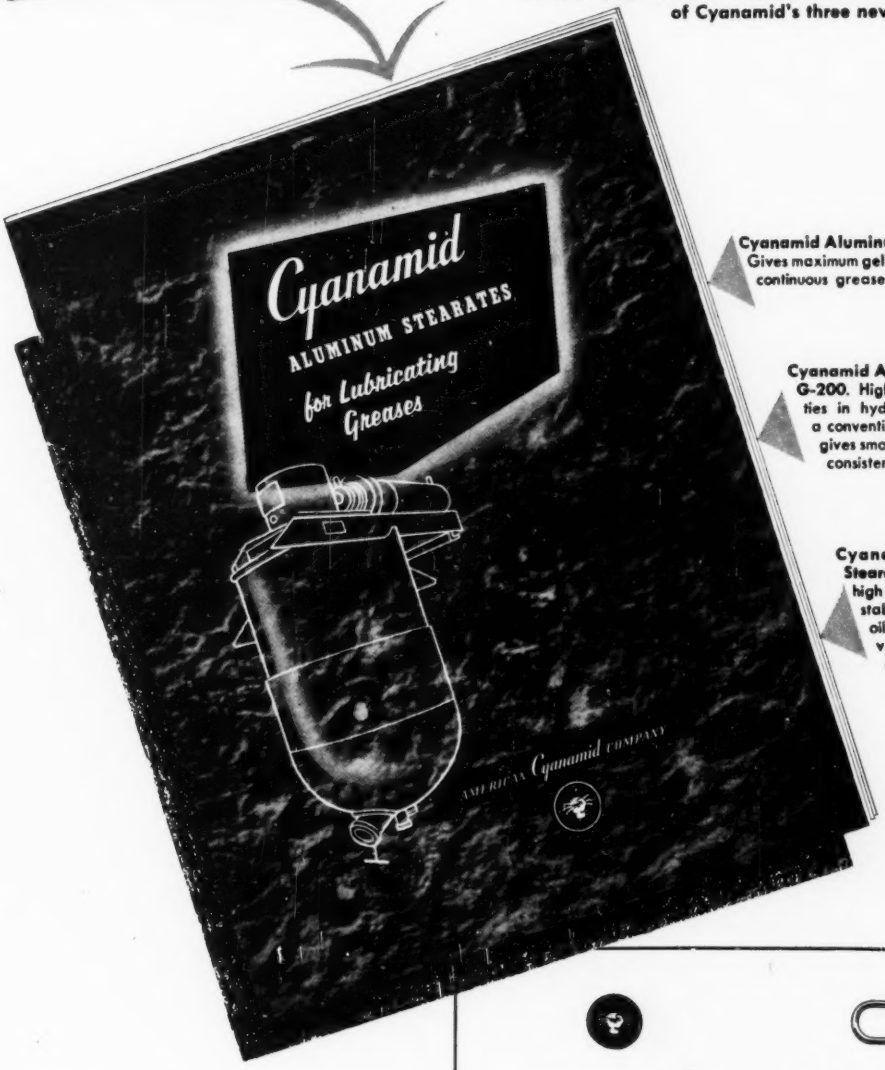

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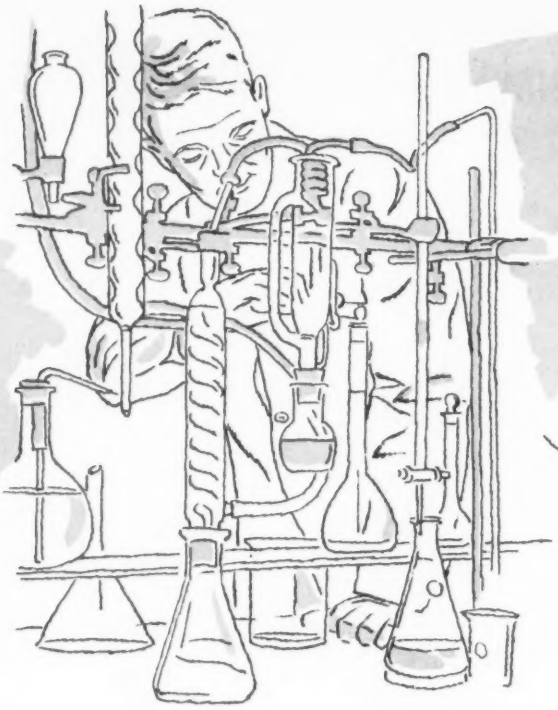

MILITARY —THEIR

I am a member of the Office of Petroleum Programs Munitions Board whose functions and responsibilities are to facilitate service and inter-service planning and coordination of effort in the field of policy and planning relating to petroleum matters. The Munitions Board Office of Petroleum Programs is further responsible for making recommendations and performing services, duties, and functions in petroleum matters, as requested by any agency of the Department of Defense. In performing its functions the Office of Petroleum Programs maintains contact with other governmental agencies and with the Military Petroleum Advisory Board, an industry advisory group.

The military services require an increasing number of specialized types of equipment, each of which makes more complex the problem of supplying satisfactory lubricants. This problem is recognized. Continued effort is being made to increase the application and temperature range over which lubricants can successfully operate. The results of these efforts are reflected in the combining of the different requirements into joint specifications and the cancellation of those specifications no longer required. A reduction in the number of products required will simplify the manufacturing problems for industry and the logistics problems for the services.

An important job for the future in connection with the development of greases and lubricating oils, of the petroleum and synthetic types, is the necessity of greater correlation between service-performance and laboratory performance tests, together with refinements of laboratory methods for greater reproducibility.

Equipment supplied by industry to the military is serviced by the manufacturer with lubricants fully meeting the requirements of the service involved. It is proper that as the needs of the service develop, industry be given complete information concerning them so that satisfactory products may be developed. This makes the exchange of information between the military services and industry increasingly necessary, a procedure which will result in increased availability in case of emergency.

The grease industry's manufacturing capacity is apparently adequate. The supply of some materials required for specialty greases may not be. The military services have experienced difficulty in obtaining from industry adequate coverage for their requirements of small quantities of special oils and greases.

Industry should appreciate the military position regarding these comparatively small volumes of special products. The requirements have been established through extensive research and sufficient quantities must be available to successfully operate the equipment for which they were developed.

Even at the expense of being repetitious it is deemed necessary to mention again where copies of petroleum specifications may be obtained. They may be procured from the Superintendent of Documents, or from any of the three services. New indexes of specifications and standards used by the services will soon be available by individual order or on a subscription basis from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Actual delivery date is uncertain at this time. Monthly

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From a military standpoint, standardization and or consolidation is a basic concept and is rightly so. Every activity within the military is striving for the utmost in this regard. Much progress has been made. As far as specifications are concerned, all the services are constantly trying to reduce the number of products required. This is done because it is militarily expedient and logistically safer to have the fewest possible items to provide in combat areas. This also is true in other areas, but not as vital. Troubles arise with the number of products because each new advance in machinery or equipment seems to present the tempting prize of superlative performance with rare lubricants. Some developments naturally require special products because of design for those products in exclusion of others. This is more frequently the case for fuels of non-petroleum nature because there seems to be some degree of flexibility of requirements for using petroleum products in most new developments.

The quality of products required is dictated by the equipment and the anticipated conditions of service or operation. Quality is always tempered by availability. Availability is a big word in specifications, even during peacetime, because the equipment must use the material which would be available in sufficient quantity during emergencies or war when, as you all know, petroleum becomes a critical item.

The aim is to ask for petroleum products of the quality consistent with requirements in military operation and with adequate supply.

The military services constantly try, with the assistance of industry, to simplify its specification and to reduce the number of tests required to establish product quality. This, as can be well appreciated, is a constant struggle because each change or improvement raises the question of correlation between laboratory tests and service. These efforts are worthwhile and the services plan to continue them.

In discussing the individual items and groups of items it can be safely said that no greater progress has been made in the consolidation of any lubricant or related specifications than in the case of the corrosion preventives. In this instance fourteen specifications have been combined into three specifications, having seven grades. Specifications AF-14156A (1), MIL-R-15074 (Ships), EES-C-3348, and AXS-1719 now compose one specification, Corrosion Preventive, Fingerprint Remover, MIL-R-15074. Air Development Force, Wright

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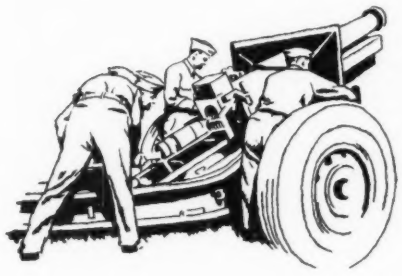
Field, is a custodian and is presently concerned in developing a list of qualified suppliers.

The second of the new corrosion preventive specifications is MIL-C-15167, Corrosion Preventive, Petroleum Type, Hot application, for which the Army is custodian. This specification supersedes AXS-1347, 2-84, 52-C-17Ty, B & C 14-C-8, AN-C-124Ty2, MIL-C-6708Ty2, 2-82 and MIL-C-15167 Ships (1). This includes three grades of pigmented



and non-pigmented material and at present is being circulated for comment. A list of qualified suppliers is being prepared.

Corrosion Preventive, solvent cutback, cold application MIL-C-16173, for which the Navy is custodian, replaces MIL-C-16173 (Ships), 52-C-18 Gr. 1, 2, & s, 14-C-9, AN-C-124Ty1, AXS-1759 Gr. 1 & 2, MIL-C-972, Ships (1), MIL-C-6708, Tyl and AXS-673. Three grades are included. The Bureau of Ships at present is in the process of coordinating this specification as well as compiling a list of qualified suppliers.

All the technical details of the three specifications described above were processed and consolidated by representatives of the Army, Navy and Air Force in a little more than a year's time. It represents a major contribution to the unification effort.

In regard to future quality requirements for corrosion preventives it may be mentioned that present thick film corrosion preventives are, in general, satisfactory but have a limited climatic range. Improved materials having satisfactory performance in arctic and tropical climates, as well as in temperate climates, and possessing greater ease of removability are definitely needed. Future specifications for thick film materials will include the previously named qualifications.

Suitable additives for thick film corrosion preventives are in short supply and would have most limited availability in times of emergency. Wholly synthetic corrosion preventive additives not dependent on by-products or natural products of limited availability need to be developed. In fact it is almost essential that they be developed since more and more of this type of material is being used and is needed for lubricating oils.

Applications of volatile corrosion inhibitors to engine and ordnance equipment are under study. The application of such inhibitors to ferrous metal parts and components will continue to increase throughout the short and long-range period. The specification for this material has been written and is now in the process of being coordinated. Also it has been agreed that qualification and approval test work will be conducted at the Naval Air Experiment Station at Philadelphia.

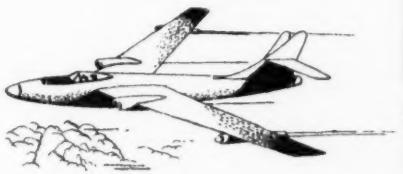
The trend in grease specifications is very markedly toward wide temperature ranges and greater oxidation stability. There is more reliance and emphasis on performance requirements. High speed and temperatures of operation and long bearing life are common words used in grease discussions these days. The research activities of the Navy have played a leading part in the development of lithium soap greases which are highly suitable for a relatively wide temperature range.

Grease specification MIL-G-3278 resulted from a combination of (AN-G-25) and (2-134) with Bu Aer as the custodian. Amendment 1 is in process.

The grease used for extreme pressure applications, AN-G-10, Low and High temperature, has become MIL-G-7188 (1) and Amendment 1 is now in process. In investigating AN-G-10, the goal desired was an extreme pressure grease with the temperature and stability characteristics of an AN-G-25 type but with as high a consistency as possible.

Lubricating Grease, General purpose, Aircraft MIL-L-7711, is the new name of the general purpose grease, AN-G-15A, since it has become MIL-L specification. The corrosion preventive properties of this specification are considered inadequate and there is still some feeling that it could be superseded by MIL-G-3278.

For some time the objective of the Army has been to develop a single all-purpose, all-temperature automotive and artillery grease which would be the principle item of



field supply. It was realized such a product would represent many compromises but was consistent with the over-all military picture. Work to date has indicated that a specification of this nature is within the realm of possibility and an uncoordinated specification has been printed. It will be coordinated as Lubricating Grease, Automotive and Artillery, MIL-E-10924 and will supersede MIL-G-10924 (Ord) MIL-G-2108, 2-108, 2-39, AXS-785, 2-106, AXS 781, AXS-1169, 14-G-10 and 14-G-3c.

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It appears that the actual consumption of fluorinated oils by the Air Force is small. There are many applications but the volume requirements are not large for each particular application.

A typical application is lubrication of the ball bearing of a rocket engine turbine pump set for white fuming nitric acid JP-3 fuel combination. This bearing will require approximately one pint per power plant per year if design is not altered. Some other applications are servicing trailers, packing gland filler and deal lubricant, and for use in the gyro units of fire control systems.

The greatest usage of the fluorine containing materials is in the form of solid polymers in uses such as coatings of tanks or wherever extended contact with nitric acid is required; as a high temperature insulation for wires and other parts in electronic equipment.

Bentone greases are under experimentation by the services but at this date none have been put in actual service.

With regard to synthetic greases the Army is continuing to survey the whole field of these materials and initiating performance tests which it is hoped will bring to light factors that will tend to improve their over-all performance. Both the Navy and Air Force are actively investigating the synthetics, but to date none have proven entirely satisfactory in all the necessary physical and operational characteristics. However, there is being conducted a study of the availability of raw materials, particularly diesters, for use in manufacturing synthetic lubricants.

It is expected that the availability of dibasic acids and alcohols will be the controlling factor in the selection of certain diesters and complex esters for large-scale production. The components which are currently being considered as most important for the manufacture of esters comprise straight chain dibasic acids with 6 to 10 carbons per molecule and branched chain primary alcohols with short alkyl branches.

Also of interest are the "complex" esters which may be blended with diesters to increase their high temperature viscosity and load-carrying ability without seriously affecting their low temperature properties. The availability of the acids and alcohols required for the complex esters is believed to be about equal to that for the most procurable diester components. Industry capacity for producing polyglycols required in certain complex esters is unknown, but the supply of raw materials, ethylene, glycol and ethylene oxide, is believed quite ample.

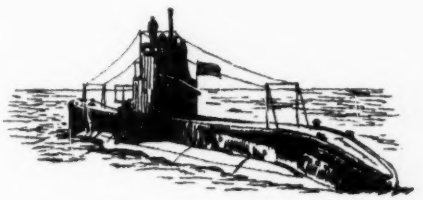
It is predicted that, if the availability study should prove favorable, research and development work on the synthetic lubricants will be stepped up considerably.

While it is true that greases for high torque aircraft equipment permitting operation between —65°F. and 300°F. are available, they are still not quite adequate. The upper temperature limit should be extended to 400°F. Greases for high and low torque equipment with —65°F. to 400°F. operating range are needed as well as greases that will perform from a temperature of —100°F. to 250°F.

While no formal complaint has been received, Navy representatives have been advised that there is room for improvement in snorkel tube lubrication. The application of cup grease to the telescoping surfaces by hand is a time-consuming operation. A grease which would stay on the tube for a long period of time would save time, might give better performance and would certainly improve crew morale in the sense that a dirty and troublesome job would be eliminated.

Under the present specifications, adequate hydraulic fluids are available for ground equipment. The disadvantage is that different fluids are required for brake systems, recoil mechanism, hydraulic actuating systems and shock absorbers. A single fluid, satisfactory for all such applications, cannot be used until satisfactory materials for packings, hoses, etc., have been adopted. In the case of aircraft, there is no non-inflammable hydraulic fluid which will meet existing requirements of all services. Neither is there a non-slick hydraulic fluid available for submarines. Assuming the development and adoption of suitable materials for packings, hoses, etc., a satisfactory fluid may be available. It should be pointed out that development should be stepped up for fluids for use from —65°F. to 400°F. for application to sonic and supersonic aircraft and missiles.

Satisfactory gear oils are available at the present time. AN-O-3 (6), Gear Lubricant, Low Temperature will become MIL-O-6086 with no changes. The Air Force will be custodian. Army Ordnance will be custodian of the new specification universal, Gear Lubricant, Sub-zero, MIL-L-10324, which will supersede MIL-L-10324 (Ord) (2). Present



arctic gear oils are satisfactory for temperatures of —65°F. but present equipment requires that all gear oils have improved corrosion prevention properties.

In aircraft equipment, turboprop reduction gear lubricants require intermittent operation at temperatures of 400°F. to 500°F. and have high load support requirements. It may be possible that diester and other type synthetic lubricants will be needed to fulfill the requirements of these applications.

On shipboard, improved lubricants may be required for the new gear reduction units now being developed as soon as the lubricant requirements for these gear units are defined.

With regard to engine lubricants, Navy specifications 14-O-13a will sometime in the future become MIL-L-9000, Lubricating Oil, Heavy Duty, Diesel. However, action has not yet been initiated on this matter. MIL-O-2104 (Ord) will be coordinated to become Lubricating Oil, Heavy Duty Engine, MIL-2-2104.

No progress is being made toward a uniform specification combining MIL-O-2104 (Ord) and Navy Symbol 9000 oils. The latest statement by the Navy is that "Adequate data is not yet available to determine the relative performance qualiy levels of MIL-O-2104 (Ord) and NS 9000 oils in Naval equipment, nor the possibility of combining the requirements for these oils into a single specification. However, data should be obtained in the normal course of brand approved testing of NS 9000 oils which have been qualified under MIL-O-2104 (Ord), and action will be initiated to seek authorization of special tests to accelerate procurement of such data."

Widespread use of synthetic lubricating oils for reciprocating engines is considered unlikely any time in the near future, although there possibly will be increasing use for special purposes and may be required for turbojet engines. These oils will be required to be operable from 65° F. to 400° F.

An improved low temperature petroleum-synthetic lubricating oil is now available for ground vehicle operation, permitting satisfactory engine starting and operation at —40° F. and not requiring dilution. MIL-O-10295 (Ord) Oil Engine Sub-Zero is the number and title of this specification.

Looking into the future a bit as far as the new Diesel engines being developed are concerned, indications are that they will be light in weight and high in specific horse power output. In addition the engines may have silver bearings. The use of silver bearings presents a real problem and currently available bench tests are not adequate to qualify lubricating oils as regards their corrosiveness to silver bearing surfaces. An oil which passes the EMD silver strip test and has shown both passing and failing performance in the Underwood OM-2 test caused serious corrosion of bearings in the 16-338 Diesel Engine in 150 hours of operation. The lubricating oil in these engines will be exposed to high operating temperatures and may be forced to be used with fuels containing one per cent sulfur or more. This will probably mean that the additive content of the oils will be increased which may complicate matters because as you all know, the sulfonate situation even at present is anything but stable.

The services are currently testing additive oils for use in heavy bomber aircraft and other aviation type engines but no figures are available as to what percentage of additives will be required. However, it is possible that the Air Force and Bureau of Aeronautics will in the future begin purchasing additive type oils for use in aviation reciprocating engines.

Specification Lubricating Oil, Engine, Aircraft, MIL-O-6082, Amendment 1, will eliminate the necessity for oil dilution of carrier type aircraft operating in arctic and subarctic climates.

It is necessary to repeat previous requests by the military in that designers and manufacturers must bear in mind that their equipment must utilize lubricants which are readily available in quality and quantity in event of an emergency.

The degree or extent of deterioration of stored lubricants and greases should be determined for each product under varying atmospheric conditions. This change with length of storage should be determined and its effort should be evaluated in terms of usabality. This basic information should be provided for all petroleum products and synthetic lubricants to allow the military services to stockpile as dictated by needs, products availability and transportation.

For both the military and industry the immediate future is the period for improvement. Techniques should be improved, composition of present products reevaluated, new materials evaluated, new products developed, and a further reduction effected in the number of necessary products.

In order to accomplish closer cooperation and exchange of information, all military information on lubricants will be made available to you to the extent that security permits.

The military appreciates your interest, support and understanding of its problems.

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Patents and Developments

ALKALI METAL SOAP GREASES FOR HIGH TEMPERATURE USE

One recent development is the incorporation, with a higher fatty acid soap, of a metal salt of a low molecular weight acid for the purpose of improving the structure as well as stability of the grease to high temperatures in the neighborhood of 350°F. or even higher. One theory of the mechanism involved proposes the formation of a complex of the two types of acids, giving greater thickening effect and high temperature stability.

In U. S. patent 2,575,286 issued to Standard Oil Development Co., the low molecular weight acids are specified as oxy acids containing an ether linkage such as ethoxy propionic acid, methoxy propionic acid, iso-propoxy propionic acid, sec-butoxy acetic acid, and the like. Ethoxy propionic acid is prepared by reacting beta propiolactone with ethyl alcohol.

An example of one type of grease made in this manner has the following formulation:

	% by wt.
Ethoxy propionic acid	**6.0**
Hydrogenated fish oil acids	**15.0**
Sodium hydroxide	**4.4**
Phenylalpha naphthylamine	**1.0**
Lubricating Oil (500SSU/100°F.)	**73.6**

When the properties of such a grease are compared with similar grease in which furoic acid, levulinic acid and caproic acid are substituted for the ethoxy propionic acid, it is found that the latter greases exhibit considerable superiority as is apparent from Table I.

It is preferred to employ about 3-20% of the high molecular weight fatty acid soap and 2-15% of alkali metal salt of ethoxy propionic acid.

TALL OIL SOAP GREASES

In U. S. patent 2,516,136, high temperature greases were disclosed employing salts of heterocylic acids with conventional soaps as thickening agents. It has been found that such greases can be produced very economically by substituting tall oil for the fatty acids in the compositions. According to the recent Standard Oil Development Co. patent 2,576,031, the furoic salts may be produced in situ by means of a Cannizzaro reaction wherein furfural or thiophene aldehyde is treated with a strong base, as described in U. S. patent 2,516,137.

Since fats and fatty acids have been difficult to obtain while tall oil is a relatively cheap by-product of the wood pulp industry, the process employing such a substitution product, is of considerable interest when a high quality grease is capable of being prepared.

The data on manufacture of a typical grease are given as follows:

CHARGING FORMULATION
Ingredients
Cannizzaro Reaction

	% by wt.
Furfural	10.00
Sodium Hydroxide	3.18
Mineral Oil, 60 S. U. S. Viscosity at 210°F.	30.00
Tall Oil	15.00
Sodium Hydroxide	2.00
Phenyl Alpha Naphthylamine	1.00
Mineral Oil (as above)	38.82

Per Cent Free Alkalinity—0.22% as NaOH.

In the manufacture of this grease, the furfural (1 part by weight) and a portion of the mineral oil (3 parts) were charged to a grease kettle and stirring initiated. A cold aqueous 33⅓% solution of NaOH (shown in the above formulae as part of the Cannizzaro reactions) was next added. After the reaction is completed as shown by subsidence of the temperature of reaction, the tall oil (having an acid number of 183.2 and Sap. No. of 185.2) was added and neutralized with the balance of the NaOH as a 40% aqueous solution.

The temperature was next raised by external heating to 400°F. while adding the balance of the mineral oil in small portions. At this temperature the phenyl alpha naphthyla-

TABLE I

	Ethoxy Propionic Acid	Furoic Acid	Levulinic Acid	Caproic Acid
Per Cent Free Alkalinity	0.12	0.06	0.27	0.25
Worked Penetration (77°F.)	306	234	358	Grease did not make.
Structural Stability (Penetration 100,000 Strokes in worker)	339	272	Semi Fluid	Soap separates out.
Dropping Point, °F.	446	488	378	
Dispersability of Soap	Excellent	Fair	Poor	
Wheel Bearing Test, 6 hours at 220°F.	Pass	Pass	Fail	

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mine, used as an antioxidant, was added and the grease was drawn into pans for cooling. It could be worked down in the kettle while cooling if desired. The cold grease was then homogenized by working in the grease kettle to a smooth, uniform product after which it was filtered and packaged. This grease contained about 6% sodium furoate and about 15% soda soap of tall oil.

The following properties were found upon inspection:

Worked penetration	180 mm./10.
Structure stability, as measured by a penetration of 268 mm./10 after working 100,000 strokes with a fine hole worker plate	Excellent.
Dropping point, °F.	Above 500.
50 hour separation test, 60 mesh cone at 210° F. (per cent separation)	None.
Wheel bearing test, 6 hours at 220° F.	Pass.[1]
Oxidation test, Norma Hoffman bomb	102 hrs. to 5 p.s.i. drop in pressure.

[1]No slumping, no oil separation, slight hardening.

HIGH TEMPERATURE FURFURAL GREASES

The use of the Cannizzaro reaction (as described in U. S. patent 2,516,137) already has been discussed in the preceding section, showing that it serves as a means for condensing furfural in a grease to give it high temperature-resisting properties. Another recent patent issued to Standard Oil Development Co. (U. S. 2,576,033) discloses that it is highly important to maintain a low cooking temperature to prevent formation of oil-insoluble and non-lubricating bodies in the grease.

In this case, about 3-25 parts by weight of heterocyclic aldehyde (having no alpha hydrogen and having a 5-membered heterocyclic ring) are added to 15-55 parts of mineral lubricating oil. Then an alkali metal base is added to form the Cannizzaro reaction products in the oil. Thereafter 3-30 parts of soap of C_{12}-C_{22} fatty material are formed in the oil which is cooked to 290°-350° F. Finally, 20-65 parts of lubricating oil are added at 400°-475° F. to complete the cooking and form a high temperature stable lubricating grease.

It appears that a fairly high molar ratio of (furoic) salt to soap is desirable where the soap content of the finished grease is 15% or more by weight. When the soap content is less, the salt content should be reduced more than proportionally.

For lubrication of anti-friction bearings, it appears that a suitable grease should retain its grease structure after severe mechanical working and should also have a consistency after moderate working of not less than about 230 and not more than 300 mm./10 penetration (after 60 strokes in the standard ASTM grease worker).

In order to avoid polymerization and other difficulties, it is preferred to use a steam kettle operating in the range of 380°-400° F.

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The effect of the cooking temperature upon the worked penetration of the grease is depicted in the cut (Figure 1). The advantage of higher cooking temperature is apparent. It will be noted that the dropping point of the soda grease was high in all cases.

NEW DEVELOPMENTS

Lubrication of wire rope—Rigby, Iron & Steel Engr. 9/51 p. 131.

Properties of fluorocarbon and polysiloxane fluids—Bondi, J. Phys. & Colloidal Chem. 10/51 p. 1355.

Laboratory screening of multi-purpose greases—Ten different types of tests used in laboratory evaluation to determine what performance will be under actual operating conditions —Kipp et al, Iron & Steel Engr. 9/51 p. 107.

Stopcock greases—Brooke, J. Chem. Ed. 11/51 p. 602.

Lubrication of enclosed gears subjected to chronic water conditions—a specially compounded solvent cut-back leaded compound found particularly effective in resisting effects of water—Rigby, Iron & Steel Engr. 11/51 p. 73.

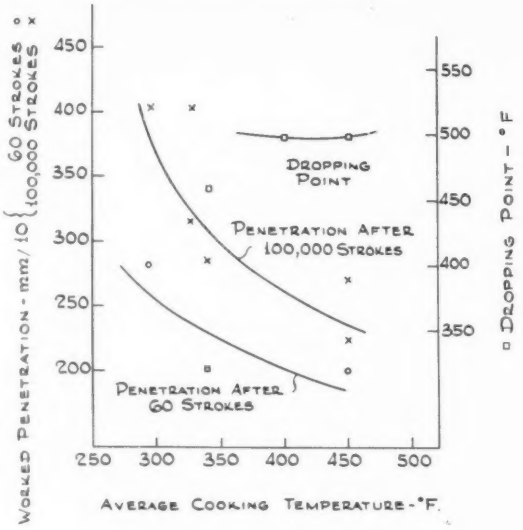
ISSUED PATENTS

Brit. 664,214 (Standard Oil Development Co.)—Lubricating grease compositions.

Brit. 664,546 (DuPont)—Making plasticized polymers and thickened oils or greases.

FIGURE 1

EFFECT OF COOKING TEMPERATURES ON FURFURAL GREASES
(10% FURFURAL BEFORE REACTION)



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WILKINSON GIVEN NEW POST WITH CONTINENTAL CAN

Lawrence Wilkinson succeeds J. S. Snelham as vice-president in charge of finance for Continental Can Company upon Mr. Snelham's retirement January 1, according to an announcement by General Lucius D. Clay, chairman of the board.

Mr. Wilkinson has been a vice-president of the company since September, 1951, prior to which time he had been director of the New York Civil Defense Commission, a colonel in the U. S. Army and a vice-president of Marine Midland Trust Company.

Mr. Snelham will continue to serve as a consultant to the chairman and president. He has been vice-president in charge of finance since October, 1950. He joined the company in 1929 as comptroller and became a vice-president and comptroller in 1936. His association with the company, however, dates back to 1916 as an outside auditor and partner in the firm of Deloitte, Plender, Griffiths and Company of New York.

LEFT, Lawrence Wilkinson fills vacancy left by J. S. Snelham, ABOVE, retired, vice-president in charge of finance for Continental Can Company.

"The contribution of Mr. Snelham to the success of Continental has been a great one," General Clay said. "Fortunately, his vast knowledge of company affairs and his wise counsel will continue to be available."

DEEP ROCK PROMOTES LEHMAN H. SULLIVAN

Lehman H. Sullivan has been promoted to assistant manager, Lubricating Oil Sales Department, Deep Rock Oil Corporation, according to W. M. Murray, Lubricating Oil General Sales Manager.

The new assistant manager has been with Deep Rock since 1937, with the exception of two years in the U. S. Navy during World War II.

Mr. Sullivan joined the company as a junior chemist in its Cushing, Oklahoma, refinery. He moved to Chicago in 1939 as assistant to the manager of the Lubricating Oil Sales Department where he remained until the beginning of World War II. After the war he returned to Deep Rock as Superintendent of Lubricating Oil Sales in the Wisconsin Area.

Two years ago Mr. Sullivan was promoted to Area Sales Manager of the Southwest District with headquarters in Tulsa and held this job until his present promotion.

Mr. Sullivan is a native of Jackson, Tennessee, and was granted a Bachelor of Science Degree in Chemistry from the College of the Ozarks. He is a member of the Society of Automotive Engineers and of the Tulsa Sales Executives Club.

PERSONNEL CHANGES MADE IN DEEP ROCK'S CONTROLLER'S DIVISION

Several personnel changes have been made in the Controller's Division of Deep Rock Oil Corp., announced A. R. Bell, Jr., controller.

Maurice B. T. Davies, formerly Manager of the Auditing Department, has been appointed manager of the Accounting Department. He is succeeded by W. Paul Hodges, who was formerly assistant manager. John W. Featherston, Jr., who was formerly Manager of the Accounting Department, becomes assistant to the controller.

Mr. Davies joined Deep Rock in July of 1949 and is a Certified Public Accountant. Mr. Featherston also joined the company in July of 1949. He is a graduate of Louisiana State University and had 12 years accounting experience in the oil industry before joining Deep Rock.

Mr. Hodges is a University of Arkansas graduate who came with Deep Rock in November of 1949 after a number of years with a Tulsa accounting firm. He is a Certified Public Accountant and secretary of the Tulsa chapter of the Institute of Internal Auditors.

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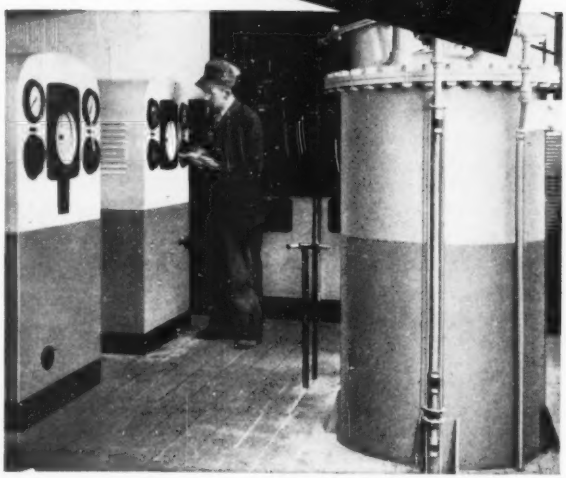

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ESMAY APPOINTED EXECUTIVE DIRECTOR OF OIIC IN ABSENCE OF H. B. MILLER

Edwin W. Esmay, of the Standard Oil Company (New Jersey), has been appointed Acting Executive Director of the Oil Industry Information Committee, to serve during the leave of absence of H. B. Miller, who has been elected President of the National Committee For A Free Europe.

Mr. Esmay also will serve as Acting Director of the American Petroleum Institute's Department of Information according to President Frank M. Porter. The dual appointment is effective March 1, at which time Mr. Miller will take over his new duties as head of the private citizens' organization whose objective is to combat world communism.

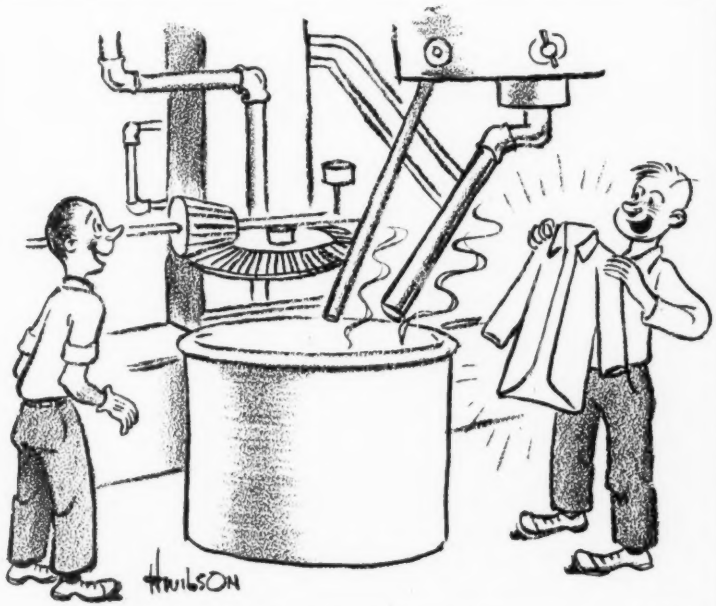
Mr. Porter announced that the temporary change in top personnel will not affect the basic operations of the Oil Industry Information Committee.

The A P I president noted that Mr. Esmay is well qualified for the temporary replacement. Mr. Esmay is a former employee of the Institute and served as Acting Director of Information in 1939 and 1940. He has been identified with the O I I C since its beginning, and served as executive assistant to the chairman from 1946 through 1949.

Mr. Esmay has been granted a leave of absence from his firm to carry on the O I I C operations. He will work closely with Charles Z. Hardwick, of Findlay, Ohio, a director and vice president of The Ohio Oil Company, who is 1952 National Chairman of the Oil Industry Information Committee.

Mr. Esmay currently is industry liaison representative in the public relations department of Standard Oil Company (New Jersey), a position he has filled since 1946. He was born and raised in Utica, N. Y. He attended

(*Continued on page* 37)



"I didn't develop that new gear lubricant—but look at this swell petroleum shirt."

NILES

Steel Pails and Drums

FOR OILS, GREASES, ETC.

No. 9 Niles Pail with lug cover and pour spout.

NILES STEEL PAILS

ordinarily are supplied with bail handles. Fabricated from black steel—outside painted or lithographed to order. Removable tops with lug covers, with or without pour spouts. Also in tight head style. 3½ to 6 gallon capacities.

• • •

NILES STEEL DRUMS

55, 30 and 15 gallon capacities; and 100 lb. grease drums. Full open head or Bung type. Painted or fully decorated. Interior lacquered if desired.

100-lb. Niles Grease Drum with 14 inch full open top lug cover. Also made with 9 inch lug cover in center of head.

NILES STEEL PRODUCTS DIVISION
REPUBLIC STEEL CORPORATION
465 Walnut Street • Niles, Ohio

Grease gun fits into grease follower plate through center opening in pail cover. Slight downward pressure on gun releases plate from cover.

No contamination . . . U-PRESS-IT cap snaps on cover opening when grease pail is not in use, providing double protection with follower plate.

No grease waste . . . as gun handle is withdrawn, grease is quickly and uniformly drawn into gun barrel.

Sturdy construction of pail, with opening recessed in top, cuts in-transit damage losses to a minimum and assures long life.

No air bubbles . . . because follower plate maintains uniform pressure on grease during gun loading.

Special sponge rubber gasket adapts itself to any dents in walls of container and sweeps sides clean as follower plate moves down pail.

Inland KLEEN-FIL Grease Gun Filler Pails available in 25 lb. (3½ gal.) and 35-40 lb. (5 gal.) sizes, lithographed to your specifications.

Pail shipped from factory with follower plate attached to cover to simplify handling during grease packing operations.

Market your grease in the trouble-free

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GREASE GUN FILLER PAIL!

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Your Industry NEWS

"FRIGISTAT" BATH

Precision's Redesigned "Frigistat" Provides Frost-Free Operation With Few Controls

Precision has completely redesigned its "Frigistat" bath to provide frost-free, efficient operation with a minimum of temperature control adjustments. Designed expressly for following A. S. T. M. Method D97, the bath, although manually operated, can easily be maintained within ± 2° F.

The 7-unit test chamber and the dry ice chamber are insulated with 2½" of glass wool at the sides and bottom. Cooling medium is circulated in the test chamber through coils which do not allow interchange of fluid between chambers. Dry ice capacity is ample. A full charge of dry ice plus the efficient ¼ H. P. pump will reduce temperature of the test chamber to —90° F. in about one-half hour.

Made of steel, except for a heavy Bakelite top, the housing is finished in Silver-tone Hamerloid baked enamel. Inner chambers are pure spun copper; all pipes and fittings are of brass. Overall dimensions: 24½" wide by 13" deep by 18" high. Immediately available. Precision Scientific Company, 3737 W. Cortland St., Chicago 47, Ill.

ATTEND API LUBRICATION COMMITTEE MEETING

SHERATON-CADILLAC HOTEL DETROIT, MICHIGAN FEBRUARY 18 & 19

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Pittsburgh, Pa.—Toledo, O.—Cincinnati, O.
Port Arthur, Tex.—Fort Worth, Tex.
Sweetwater, Tex.

William H. Oldacre

Too sudden to be realized or fully grasped came the tragic news on Friday, January 18, that William H. Oldacre had unexpectedly passed away. President and General Manager of the D. A. Stuart Oil Company for the past 32 years and active in many technical societies, his untimely death spelled the end of an active life of leadership in the petroleum industry.

Prominent in NLGI affairs since its early years, he had served on many committees. In 1943 he was vice-president and became president in 1945. For the past several years he was co-chairman of the Technical Committee. During the past year he was a member of the Membership Committee and had continuously served the lubricating grease industry as a member of our board of directors since 1936.

Prior to becoming President and General Manager of D. A. Stuart Oil Company, Ltd., he had been Director of Research for that organization.

His specialized interests lay in the field of metal cutting leading to his outstanding knowledge on the subjects of sulphurized and sulpho-chlorinated oils and lubricating greases. His early background leading to these interests lay in his engineering experience with the General Electric Company and Timken Roller Bearing Company, which introduced him to the oil business. Prior to his early experience with them he attended and was a graduate of Hiram College and became an honorary member of Pi Tau Sigma, national mechanical engineering fraternity.

The lubricating grease industry knew him as a man who thought largely on industry subjects evidenced by his membership in a number of engineering and technical societies including the American Society of Mechanical Engineers, Society of Automotive Engineers, American Petroleum Institute, National Lubricating Grease Institute, American Society for Lubrication Engineers, American Society for Testing Metals, American Society for Metals, American Society of Tool Engineers, American Chemical Society, American Iron and Steel Engineers, Independent Research Committee on Cutting Fluids and the Coordinating Research Council.

ESMAY APPOINTED . . .

(*Continued from page* 33)

Princeton University, and was associate editor of Fueloil Journal from 1933 to 1937, at which time he joined the staff of the American Petroleum Institute. He was Acting Director of Information in 1939 and 1940. From 1941 to 1943, he was publicity assistant to the chairman of the Petroleum Industry War Council in Washington, and public relations director of District I, Petroleum Administration for War, for a brief period. He served with the U. S. Navy in the Pacific in 1943, 1944 and 1945. In 1946, he joined Standard Oil Company (New Jersey) in New York.

Mr. Miller has been director of the A P I Department of Information and executive director of the O I I C since June, 1948.

Mr. Miller, a graduate of the U. S. Naval Academy, class of '24, became the youngest admiral in the history of the U. S. Navy during World War II. He served as a fighter pilot aboard five different aircraft carriers; commander of Patrol Squadron 33 in Panama, flag secretary to the Commander Support Force, North Atlantic; naval attache for air at London; public relations officer to Fleet Admiral Nimitz during the Iwo Jima and Okinawa campaigns; and director of public information for the Navy Department.

RAINEY JOINS FOOTE AS MINING ENGINEER

Dudley L. Rainey has recently joined the Foote Mineral Company as Mining Engineer of the company's Kings Mountain, North Carolina branch. Mr. Rainey is a graduate of the Colorado School of Mines. Prior to his joining the Foote staff he was associated with Columbia University as Research Assistant doing research in mining methods. Mr. Rainey is a member of the American Institute of Mining and Metallurgical Engineers.

NATIONAL LEAD NAMES W. T. TRASK AS MANAGER OF STEEL PACKAGE DIVISION

Warren T. Trask has been named manager of National Lead Company's Steel Package Division. Mr. Trask was employed in 1946 as manager of steel package sales in connection with the expansion of operations of the Steel Package plant in Granite City, Illinois. Since April of 1950 he has been manager of steel package sales for the company. Prior to coming to National Lead, Mr. Trask was connected with Geuder, Paeschke & Frey Company in Milwaukee, where he was in charge of sales and administration of that company's Steel Container Division.

(*Continued on next page*)

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HOMOG-A-RATED
LITHIUM
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Our large, VERSATILE, modern manufacturing facilities are daily producing for PROMPT shipment, the following Lithium-Based lubricants
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A HIGHLY CONTROLLED COMMERCIALLY PROVED PRODUCT . . . 5400 5405
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API LUBRICATION COMMITTEE WILL HOLD MEETING IN DETROIT

The third Detroit Meeting of the API Lubrication Committee presents a well balanced program that is attracting interest from oil and automotive industries. Chairman B. G. Symon is to be congratulated in developing an outstanding contribution to the lubrication industry, which will be held at the Sheraton-Cadillac Hotel.

Scheduled at 3:00 p. m. on Sunday, February 17, 1952, will be a meeting of the Operating Committee. On Monday, February 18, the morning sessions open with registration at 10:00 a. m. followed by morning speakers: John C. Hale and G. T. Doman.

Mr. Hale, Assistant to President and Director of Maintenance, Refiners Transport & Terminal Corporation, Detroit, Michigan, has as his subject—"Quality Lubricants as an Essential to Fleet Maintenance Programs."

He will be followed by Mr. Doman, National Service Manager, Ford Motor Company, who will present the subject, "Service Approach to Customer Satisfaction."

An open session is announced for the afternoon with M. E. St. Aubin, Director, Service Section, General Motors Corporation opening the session. He will discuss "Planning and Manning for the Job Ahead."

The second speaker in the afternoon session will be C. W. Georgi, Vice-President, Quaker States Oil Refining Corporation, presenting: "Some Automotive Lubrication Problems from an Oil Marketer's Viewpoint." Mr. Georgi is well remembered for his outstanding contributions to the lubricatioon industry as a member of the Board of Directors and President of NLGI. He is author of a number of publications including his most recent book, "Motor Oils and Engine Lubrication."

The final speaker will be C. G. A. Rosen, Sonsulting Engineer, Caterpillar Tractor Company. His subject: "Co-operation Betweet Two Industries."

That evening at 7:00 p. m. will be the banquet addressed by Arthur H. Motley, fireball President of Parade Publications.

The final session will be held on Tuesday, February 19, and will be devoted to a business meeting during both the morning and afternoon sessions.

W. F. LEONARD RESIGNS AS ASLE SECRETARY

Unexpectedly came the announcement of the resignation of W. F. Leonard, Secretary of the American Society of Lubrication Engineers since 1947. Also announced was the resignation of his wife, Mrs. Patricia Leonard, Editor of their publication, LUBRICATION ENGINEER.

Leaving the Society on March 1 they plan to go to Florida on the 4th with plans to have a vacation for from 6 weeks to 2 months.

DEEP ROCK PROMOTES HARRY DERENIUK

Harry Dereniuk has been promoted to Chief Primary Engineer of Deep Rock Oil Corporation, C. A. Houy, Vice-President, has announced.

The new Chief Primary Engineer was formerly District Engineer in the Company's Wichita Falls, Texas, office. In his new assignment, Mr. Dereniuk will be located in the Tulsa general office.

Mr. Dereniuk is a native of Saskatchewan, Canada, and a Petroleum Engineering graduate of the University of Oklahoma. During World War II he served as a Flight Lieutenant in the Royal Canadian Air Force.

Before joining Deep Rock he was in charge of the Engineering Department of a southwestern gas company. Mr. Dereniuk has had considerable field experience in Texas, Kansas and Canada.

ULTRA-PENN APPOINTS RICHARD G. KEPPLER

Richard G. Keppler has been appointed Manager in Charge of Production of the Ultra-Penn Refining Company, Bruin, Pennsylvania, a wholly-owned subsidiary of the Borne, Scrymser Company.

Mr. Keppler will continue his duties as Chief Chemist of the parent company

WITCO ELECTS TWOMBLY TO DIRECTORSHIP

At a recent meeting of the board of directors of Witco Chemical Company in Chicago, W. F. Twombly, Vice-President, was elected to a directorship.

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Look at these Construction Features

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1. **Flush Ball Check**—Head can be wiped clean without forcing dirt into fitting and into bearing.
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3. **Special Spring Design**—Spring cannot be compressed to restrict flow of the heaviest lubricants.
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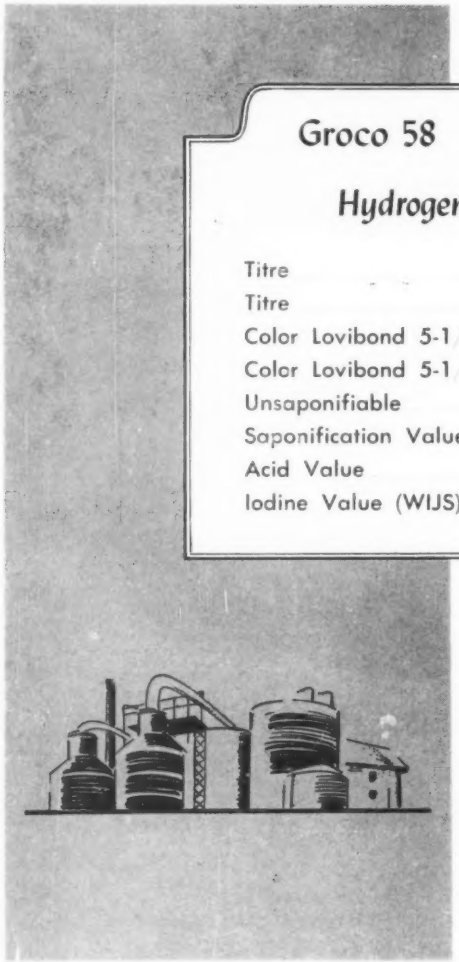

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This dictionary and handbook contains over 6,000 non-technical entries and 1,000 illustrations, especially prepared for buyers, sellers, salesmen and consumers of petroleum products. It covers the automotive and agricultural fields, also textiles, mining, lumbering, steel, railroading, ceramics, refrigeration, paper, the food and many other branches of industry.

Hundreds of lubricating and other oils are defined. More than a score of varieties of gasoline are described. The definitions of all additives generally used in present day fuels and lubricants are found here. All of the newly proposed types, definitions, designations and the functions of cutting fluids are listed. The three fundamental mechanisms employed in fluid drives are defined and illustrated by a step by step description.

Hundreds of entries define greases, their manufacture, properties, service characteristics and uses. Clear definitions of the five new API service classifications for automotive engine oils adopted by the American Petroleum Institute in November, 1951, are included. There is a definition of every term on which you would ordinarily want information in connection with the origin, history, drilling and production of crude oil. There are definitions of practical, every day terms in connection with: asphalt, fuel oils, solvents, insecticides, medicinal oils, specialties, rubber, nylon, and other synthetic products made from petroleum; gears, gear actions, gear lubricants, gear failures, etc.; bearings; distillation, cracking and refining processes; natural gas, LP-Gas, refinery gas and others.

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FEB. 18 & 19

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G. P. & F. has the experience and facilities to produce a wide variety of steel containers that will give your product full protection . . . get it to your customers safely. Every type of G. P. & F. container is designed right . . . built right, for the purpose intended. The utility values of the specialty containers add extra sales appeal to your product.

G. P. & F. Steel Containers are finished in solid colors, or lithographed with your own design. Hot dipped galvanized, tinned or terne coatings are also available. Can be ordered in straight carloads, mixed carloads or smaller quantities.

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Half Bushel Basket—Hot dipped galvanized, 30-lb. capacity.

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10⅜" Diameter Grease Pail—25-lb. size.

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FUTURE MEETINGS of Your Industry

FEBRUARY, 1952

18-19 American Petroleum Institute (Division of Marketing, Lubrication Committee meeting), Hotel Book-Cadillac, Detroit, Mich.

18-20 Oil Industry Information Committee, The Plaza Hotel, San Antonio, Texas.

19-21 Iowa Independent Oil Jobbers Assn. (annual convention), Fort Des Moines, Des Moines, Iowa.

26-27 Wisconsin Petroleum Assn. (26th annual convention and equipment show), Milwaukee Auditorium, Milwaukee, Wisc.

MARCH, 1952

3-5 Mfrs. Standardization Socy. of Valve & Fittings Industry(annual meeting), Hotel Commodore, New York, N. Y.

3-7 American Socy. for Testing Materials (spring meeting), Hotel Statler, Cleveland, Ohio.

4-6 Socy. of Automotive Engineers (national passenger car, body and materials meeting), Hotel Book-Cadillac, Detroit, Mich.

5-7 American Petroleum Institute (Division of Production, Southwestern district), Washington-Youree, Shreveport, La.

11-13 Illinois Petroleum Marketers Assn. (annual convention), Hotel Sherman, Chicago, Ill.

12-13 Texas Oil Jobbers Assn., Inc. (annual spring convention and refiners & suppliers exhibit), Hotel Adolphus, Dallas, Tex.

18-20 Ohio Petroleum Marketers Assn. (annual convention and marketing exposition), Deshler-Wallick Hotel, Columbus, Ohio.

19-21 American Petroleum Institute (Division of Production, Mid-Continent district), Hotel Broadview, Wichita, Kans.

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FUTURE MEETINGS

27-28 Texas Independent Producers & Royalty Owners Assn. (6th annual meeting), Hotel Texas, Fort Worth, Texas.

31 to Apr. 2 Western Petroleum Refiners Assn. (annual meeting), The Plaza Hotel, San Antonio, Tex.

APRIL, 1952

1-4 Greater New York Safety Council, Inc. (22nd annual safety convention & exposition), Hotel Statler, New York, N. Y.

2-4 American Petroleum Institute (Division of Production, Eastern district), Hotel William Penn, Pittsburgh, Pa.

7-9 American Society of Lubrication Engineers (7th annual meeting and lubrication show), Hotel Statler, Cleveland, Ohio.

16-18 National Petroleum Association (semi-annual meeting), Hotel Cleveland, Cleveland, Ohio.

21-23 American Petroleum Institute (Division of Transportation, products pipeline conference), The Blackstone, Fort Worth, Texas.

21-24 American Petroleum Institute (safety & fire protection committees), Hotel Texas, Fort Worth, Texas.

21-24 Socy. of Automotive Engineers (national aeronautic meeting and aircraft engineering display), Hotel Statler, New York, N. Y.

24-25 American Petroleum Institute (Division of Production, Rocky Mountain district), Gladstone Hotel, Casper, Wyo.

28-30 American Oil Chemists' Socy. (spring meeting), The Shamrock, Houston, Texas.

MAY, 1952

12-15 American Petroleum Institute (Division of Refining, 17th midyear meeting), St. Francis Hotel, San Francisco, Calif.

14-19 National Tank Truck Carriers, Inc. (4th midyear meeting), del Coronado Hotel, Coronado, Calif.

15-16 American Petroleum Institute (Division of Production, Pacific Coast district), The Biltmore Hotel, Los Angeles, Calif.

18-19 American Petroleum Institute (Division of Marketing, midyear meeting), The Copley Plaza, Boston, Mass.

21-23 Oil Industry Information Committee, St. Francis Hotel, San Francisco, Calif.

JUNE, 1952

1-6 Socy. of Automotive Engineers (summer meeting), Ambassador and Ritz-Carlton, Atlantic City, N. J.

5-6 Pennsylvania Grade Crude Oil Assn. (annual meeting), Hotel William Penn, Pittsburgh, Pa.

8-12 Canadian Gas Assn., Chateau Frontenac, Quebec City, Quebec, Canada.

9-10 Chemical Specialties Mfrs. Assn. (38th midyear meeting), Hotel Statler, Detroit, Mich.

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FUTURE MEETINGS of Your Industry

JUNE, 1952

9-13 National Fire Protection Assn. (annual meeting), Hotel Statler, New York, N. Y.

9-14 American Petroleum Institute (Division of Production, mid-year standardization conference), Brown Palace Hotel, Denver, Colo.

19-20 American Management Assn. (general management), Waldorf-Astoria, New York, N. Y.

23-27 American Socy. for Testing Materials (annual meeting), Hotel Statler, New York, N. Y.

23-27 American Inst. of Electrical Engineers (summer general meeting), Nicollet Hotel, Minneapolis, Minn.

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AUGUST, 1952

11-13 Socy. of Automotive Engineers (national West Coast meeting), Fairmont Hotel, San Francisco, Calif.

19-22 American Inst. of Electrical Engineers (Pacific general meeting), Westward Ho, Phoenix, Ariz.

SEPTEMBER, 1952

9-11 Oil Industry Information Committee, The Traymore, Atlantic City, N. J.

9-11 Socy. of Automotive Engineers (national tractor meeting), Hotel Schroeder, Milwaukee, Wisc.

10-12 National Petroleum Assn. (50th annual meeting), The Traymore, Atlantic City, N. J.

22-24 American Trade Assn. Executives (annual meeting), Royal York Hotel, Toronto, Ontario

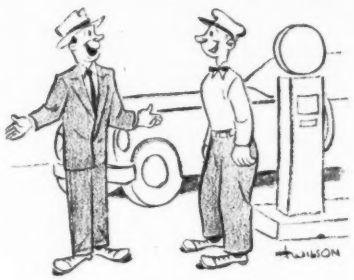
OCTOBER, 1952

1-4 (tentative) Socy. of Automotive Engineers (national aeronautic meeting and aircraft engineering display), New Hotel Statler, Los Angeles, Calif.

5-8 Controllers Inst. of America, Hotel Statler, Detroit, Mich.

9-10 California Natural Gasoline Assn. (27th annual fall meeting), The Ambassador, Los Angeles, Calif.

13-17 American Institute of Electrical Engineers (fall general meeting), New Orleans, La.



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19 Natural Gasoline Assn. of America (southern regional meeting), The Blackstone, Tyler, Texas.

20-22 American Oil Chemists' Socy. (fall meeting), Netherlands Plaza Hotel, Cincinnati, Ohio.

20-24 National Safety Council (40th national safety congress and exposition), Conrad Hilton Hotel, Chicago, Ill.

22-24 Socy. of Automotive Engineers (national transportation meeting), Hotel William Penn, Pittsburgh, Pa.

Wk.of Oct.27 American Gas Assn. (annual convention), Auditorium, Atlantic City, N. J.

28-30 American Inst. Electrical Engineers (district No. 2 meeting), Commodore Perry Hotel, Toledo, Ohio.

27-29 National Lubricating Grease Institute (20th annual meeting), Edgewater Beach Hotel, Chicago, Ill.

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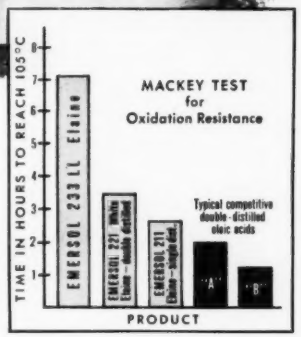
EMERSOL OLEIC STABILITY

The Mackey Test measures the auto-oxidation tendencies of oleic acid . . . giving proof of the superior oxidation stability of Emersol Elaines, as indicated by longer times.



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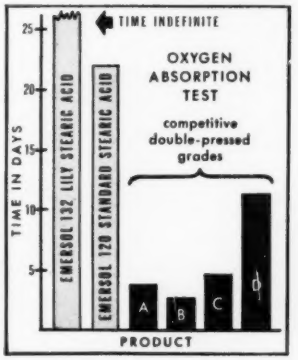

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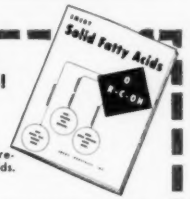

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